Hydrogen Absorption in Fluids: An Unexplored Solution for Onboard Hydrogen Storage

G. D. Berry

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FY04 LDRD Final Report
Hydrogen Absorption in Fluids:
An Unexplored Solution for Onboard
Hydrogen Storage
LDRD Project Tracking Code: 04-ERD-075
Gene Berry, Principal Investigator

Introduction

Adoption of hydrogen (H_2) vehicles has been advocated for decades as an ecological ideal, capable of *eliminating* petroleum consumption as well as tail-pipe air pollution and carbon dioxide (CO_2) from automobiles. Storing sufficient hydrogen fuel onboard still remains a great technological challenge, despite recent advances in lightweight automotive materials, hybrid-electric drivetrains and fuel cells enabling 60-100 mpg equivalent H_2-fueled automobiles. Future onboard hydrogen storage choices will be pivotal, with lasting strategic consequences for the eventual scale, shape, security, investment requirements, and energy intensity of the H_2 refueling infrastructure, in addition to impacts on automotive design, cost, range, performance, and safety.

Multiple hydrogen storage approaches have been examined and deployed onboard prototype automobiles since the 1970's. These include storing H_2 as a cryogenic liquid (LH_2) at temperatures of 20-25 Kelvin, compressing room temperature H_2 gas to pressures as high as 10,000 psi, and reversible chemical absorption storage within powdered metal hydrides (e.g. $LaNi_5H_6$, $TiFeH_2$, MgH_2, $NaAlH_4$) which evolve H_2 when warmed. Each of these approaches face well-known fundamental physical limits (thermal endurance, volume, and weight, respectively).

This report details preliminary experiments investigating the potential of a new approach to H_2 storage: absorption in fluids, specifically liquid nitrogen (LN_2). N_2 was chosen for this study because it offers unique advantages as an inert but lightweight solvent with high hydrogen solubility and is an abundant atmospheric component. H_2 absorbed in liquid nitrogen (LN_2) can be lighter than metal hydrides, with greater thermal endurance than cryogenic H_2 or LH_2, while being more compact than ambient compressed H_2.

Previous researchers[1-4] have examined H_2 mixed with a variety of simple molecular fluids (N_2, Ar, CH_4, CO). These studies were mainly aimed at the general problem of fluid phase equilibria of H_2 mixtures, and focused on identification and prediction of fluid/liquid phase boundary pressures and temperatures. In contrast, the present experiments are aimed at measuring the PVT properties of H_2/N_2 mixtures with a view toward evaluating the applicability of these mixtures for onboard automotive H_2 storage.

To our knowledge, the experiments conducted for this project are the first systematic density measurements of H_2/N_2 mixtures at cryogenic temperatures. H_2/N_2 mixtures

containing 50, 60, and 70% mole fraction H_2 were examined at temperatures of 77 K, 87 K, and 273 K, under pressures ranging from 500 to 30,000 psi (from 34 to 2000 atm), corresponding to molar densities of 15-30 moles per liter.

Experimental Procedure

The experimental study focused on investigating PVT relationships for mixtures containing 50-70 mol% H_2. This composition range is consistent with lightweight automotive hydrogen storage targets, and spans fluid behavior for H_2/N_2 molecular ratios of 1:1 to 2.3:1, exploring both "liquid-like" and "gas-like" fluid behavior. This composition range also spans the critical mixture compositions measured in the most thorough previous study[5] of H_2/N_2 mixtures at temperatures below the critical point of N_2 (126 K).

The experimental approach was designed to measure the molar density of mixtures as accurately as possible, with a critical experimental focus on maximum comparability between densities of a given mixture at different temperatures and pressures. Thus all measurements were made in the same vessel (internal volume 45.5 cm^3), allowed to come to thermal equilibrium while immersed successively in ice-water (273.15 K), boiling liquid nitrogen (77.4 K), then boiling liquid argon (87.3 K), and finally returned again to ice-water temperatures (273.15 K) to verify no gas had leaked and pressure transducer hysteresis had not occurred during the temperature cycle. Mixture pressures were observed to stabilize rapidly (typically < 5 minutes) during the experiments and were observed to remain constant for times as long as 12 hours, indicating a rapid approach to mixture equilibrium. The experimental schematic is shown in Figure 1.

Pressures were measured using a Model 2403 bonded strain gage pressure transducer from Taber Industries (North Tonawanda, NY). The transducer was certified to precision within 0.1% of full range (30,000 psi) with a calibration traceable to the National Institute of Standards and Technology (NIST). Electronic drift in the transducer was observed during experimental runs to be +/-15 psi. These experimental uncertainties were comparable to ~5% or less of the lowest absolute pressures (~600psi) measured under cryogenic conditions in these experiments, and typically <1% for higher density mixtures. The pressure transducer calibration sheet is included at the end of this report.

An independent check of experimental volume was conducted using a NIST traceable calibrated load cell scale to weigh the experimental vessel as it was pressurized with increasing amounts of pure H_2 and helium gas. The results confirmed separate measurement of the experimental volume using water. The same weighing procedure with H_2/N_2 mixtures at room temperature or in an ice-water bath (Fig. 2) verified that fluid properties software[6] (NIST REFPROP Version 7.1) provided accurate PVT calculations of H_2/N_2 mixtures across the composition range of this study for pressures up to 30,000 psi *near ambient temperature* (273-295 K).

Compositional integrity was maintained by measuring mixture pressures initially at low density, adding gas incrementally to the preexisting mixture at ~ 273 K to reach the next

higher density in preparation for pressure measurements. After recording the pressure of the highest density mixture, ~50% of the experimental mixture was captured in a sampling vessel (1 liter), yielding a cumulative (i.e. over the entire experimental run of many thermal cycles at increasing densities) mixture composition, later verified by mass spectroscopy.

The mixtures used throughout the experiments were premixed by Matheson Tri-Gas Inc. as nominal 50%, 60%, and 70% mole fraction H_2 mixtures certified to +/- 2% tolerance with the balance of the mixtures composed of nitrogen. During an experiment the gases were withdrawn and compressed from a 2000 psi bottle containing approximately 30 moles of mixture, sufficient to complete a reference run (i.e. with the experimental vessel on a balance) and an entire experimental run (pressures through cryogenic cycling) using only one bottle. Post experiment mass spectroscopy of the 50 mol% H_2 and 60 mol% H_2 mixture samples yielded actual compositions of 47.5% H_2, and 58.0% H_2, with a balance of N_2 and impurities below detectable levels (<0.1%).

In the case of the nominal 70 mol% H_2 mixture, a post experiment leak in the sampling vessel prevented accurate mass spectroscopy results. Fortunately, strong agreement between the reference run in ice water (Fig. 2) and REFPROP calculations at 273 K minimized compositional uncertainty. The gravimetric precision of the load cell scale (e.g. +/- 0.1 grams for changes in vessel weight of ~10 grams when filled with 70% H_2/N_2), combined with the large changes in mixture molecular weight for small variations in H_2 content, were consistent with an inferred mixture composition of 70 +/- 0.5 mol% H_2. The mixture composition certification for the nominal 70%/30% H_2/N_2 mixture is attached at the end of this report.

Experimental Results

The raw experimental data are given in Table 1. These include the pressure measurements for each mixture at ice-water temperatures and pressure measured after cooling to cryogenic temperatures by submerging the vessel first in boiling LN_2 and subsequently liquid argon. The associated mixture densities for a given temperature cycle were calculated for 273.15 K using REFPROP.

Table 1. Measured Pressure Data for Hydrogen-Nitrogen Mixtures

Hydrogen Fraction (of H_2/N_2 mixture)	Calculated Density (Moles/Liter)	Pressure at 273.15 K (psi)	Pressure at 87.3 K (psi)	Pressure at 77.4 K (psi)
47.5%	15.19	6745	985	845
47.5%	18.58	9438	1265	1083
47.5%	21.57	12590	1625	1380
47.5%	24.00	15900	2130	1715
47.5%	24.63	16885	2265	1790
47.5%	25.18	17795	2540	1905
47.5%	25.85	18966	2743	2035
47.5%	27.26	21695	3385	2407
58.0%	15.34	6840	1140	980
58.0%	18.51	9240	1490	1265
58.0%	21.58	12233	1981	1596
58.0%	25.85	17900	3063	2350
58.0%	27.35	20436	3473	2776
70%	8.02	2977	663	563
70%	14.15	6070	1186	1017
70%	18.93	9429	1704	1455
70%	22.33	12590	2301	1872
70%	24.73	15340	2960	2270
70%	33.10	29833	7880	6464

The measured pressures and calculated molar density of each mixture at three temperatures (77, 87, and 273 K) are plotted in Figure 3. The data for cryogenic temperatures only are replotted for greater clarity in Figure 4. Figures 3 and 4 show compositional variations between 47.5 mol% H_2 and 70 mol% H_2 had very little effect on pressure (for a fixed density) near room temperature. H_2/N_2 mixtures with higher H_2 content did have detectably lower pressures.

Upon cooling to cryogenic temperatures (Figure 4) the effect of composition on mixture pressure was reversed. Mixtures with higher H_2/N_2 ratios had higher pressures. Mixture pressure was also a stronger function of composition at cryogenic temperatures. A "knee" in density vs. pressure is discernible as mixture densities approach the density of LN_2 (28.8 mol/liter at 77.4 K). This knee is more evident when the data are plotted in terms of molar density vs. compressibility factor (Z=PV/RT) in Figure 5.

Use of the compressibility factor allows comparison of PVT properties in the context of corresponding states for any fluid (e.g. Z=1 for dilute ideal gas, Z~ 0.3 for a fluid at its critical point). Lower Z is advantageous from a storage application perspective, since this implies lower pressures or smaller volumes to store a given amount of hydrogen at fixed temperature. Figure 4 indicates that while H_2/N_2 mixtures were measured to be *less*

compressible (higher Z) than pure H_2 gas near room temperature (273 K), mixtures were *more* compressible than H_2 gas at cryogenic temperatures. Pure H_2 has a compressibility factor Z of 0.95-1.05 consistent with dilute ideal gas behavior where attractive forces between H_2 molecules play only a minor role. For mixture densities as low as 15 moles/liter, van der Waals forces reduced the compressibility factor Z significantly, to as low as 0.6 to 0.8 for H_2/N_2 mixtures containing 47.5 to 70 mol% H_2. Z increased with increasing H_2 mole fraction, and increased slightly with temperature from 77 K to 87 K.

As molar density increases, Z increased for both pure hydrogen and hydrogen-nitrogen but the relative compressibility advantage of H_2/N_2 mixtures (lower Z) was maintained up to densities of 27 moles/liter. Reducing temperature from 87 K to 77 K reduced Z the most for higher density mixtures, and improves the Z comparison relative to pure H_2 gas at constant temperature. *This suggests the most advantageous hydrogen fluid absorption storage temperature will be below 77 K both in absolute terms and relative to pure H_2 gas, even for high density H_2/N_2 mixtures.*

Figure 6 shows the volumetric hydrogen storage capacity (i.e. kg H_2/m^3) vs. pressure for each mixture at 77 K and 87 K. As might be expected, higher hydrogen mole fraction mixtures contained higher hydrogen per unit volume, however, the 58 mol% H_2 mixture stored hydrogen at densities approaching the 70% mixture. The results in figure 6 suggest that ideal H_2/N_2 mixture compositions for onboard storage will be 60 mol% H_2 or greater. Storage of 35 kg H_2/m^3 in a 70% H_2 mixture at 77 K under a pressure of only 2300 psi is a favorable result and likely represents a near optimal region of density and pressure conditions. The DOE has set a volumetric hydrogen storage goal of at least 45 kg H_2/m^3 for 2010. Figure 6 shows this was achieved at pressures of 6000 psi at 77 K for mixtures containing 70% H_2.

Comparison of REFPROP calculations with results at cryogenic temperatures

Figure 7 shows REFPROP calculations of hydrogen storage capacity vs. pressure for an H_2/N_2 mixture containing 70% mol% H_2 at temperatures of 87 K, 77 K, and 63 K. Experimental results from Table 1 are included for comparison as well as additional data points measured in a subsequent experiment surrounding the experimental vessel with LN_2 subcooled by vacuum pumping to ~ 63 K (inferred from the appearance of solid N_2).

The experimentally determined cryogenic H_2 capacities approached agreement with REFPROP calculations only at the highest experimental temperatures (87 K) or pressures (above 4000 psi). These conditions correspond to the single phase region of the H_2/N_2 phase diagram[5]. REFPROP calculations were also in agreement with experimental results for the lowest measured pressures (~500-600 psi) where the mixtures were quite dilute. REFPROP calculations overestimated experimental hydrogen capacity results for intermediate density mixtures, moderate pressures, and at colder temperatures (especially 63 K) where accurate representation of attractive van der Waals forces is especially critical (and perhaps quantum effects in the case of H_2 molecules[7]).

Figure 7 also indicates that mixtures with 70% H_2 have hydrogen storage capacity equivalent to ~80% of pure H_2 gas at 1300 psi and 63 K. This mixture contains between 30-42 kg H_2/m^3 as the pressure is raised from 1300 psi to 3000 psi, also at approximately 63 K.

Conclusions

1) The experiments identified H_2/N_2 mixtures containing more than 60 mol% H_2 and temperatures below 77 K as the most promising for H_2 storage.

2) The experiments also demonstrated that 70 mol% H_2 mixtures met the 2010 DOE onboard hydrogen storage density goal of 45 kg H_2/m^3 at 77 K under a pressure of ~6000 psi. The experiments also show that, due to the favorable compressibility factor of these mixtures, H_2 storage density decreased by less than 10% when pressure was lowered by one-third (from 6000 psi to 3800 psi).

3) 70 mol% H_2 mixtures achieved substantial hydrogen storage capacities (~30 kg H_2/m^3), even at exceptionally low pressures (1300-1800 psi) for temperatures between 63 K and 77 K.

4) As 50-70 mol% H_2 mixtures were cooled to cryogenic temperatures (77 K) measured pressures dropped by factors ranging from 5 to 10 and quickly (~5 minutes) stabilized, indicating rapid H_2 absorption and mixing with cryogenic N_2.

5) The REFPROP software from the National Institute of Standards and Technology (NIST) was found to give good agreement with experimentally determined PVT properties for room temperature mixtures and very dilute cryogenic mixtures.

6) REFPROP calculations were somewhat accurate for mixtures at 87 K, and for very compressed (e.g. single phase) H_2/N_2 mixtures, but did not adequately account for attractive forces between molecules for mixtures of intermediate density (with favorable storage pressures) at temperatures of 77 K and below.

7) Mixtures were found be significantly more compressible than pure H_2 gas of equivalent molar density at temperatures of 87 K and below, indicating stronger attractive forces between molecules in mixtures than in pure H_2 gas.

experimental effort, and enabled the experiments that were conducted to be far more fruitful.

References

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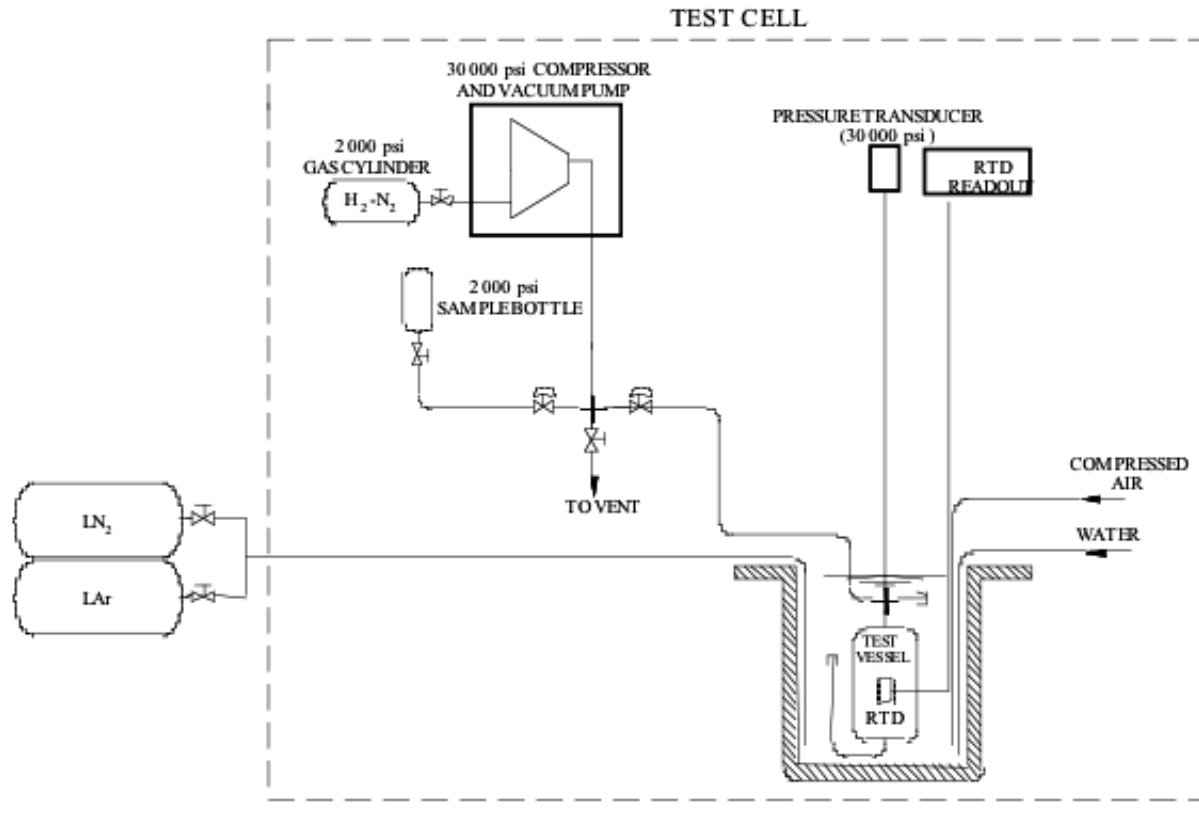
Figure 1. Experimental schematic showing hydrogen-nitrogen mixtures at 2000 psi compressed up to 30,000 psi in a test vessel within an insulated dewar. Submersion of the test vessel in ice-water (273 K), boiling liquid nitrogen (77 K) and boiling liquid argon (87 K) achieved consistent temperatures for pressure transducer measurements up to 30,000 psi. Compressed air injection rapidly evaporated liquid nitrogen and argon enabling rapid thermal turnaround.
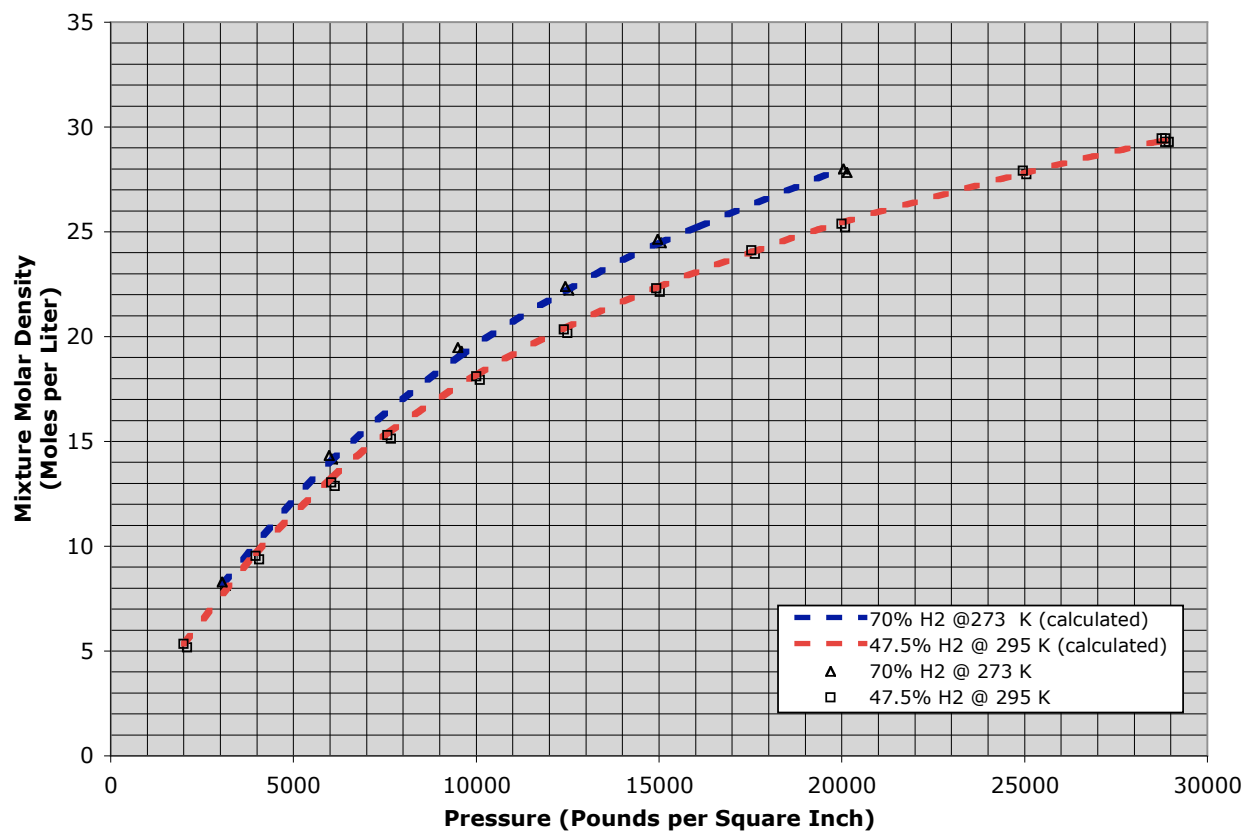
Figure 2. Molar density of hydrogen-nitrogen mixtures vs pressure as determined by weight changes in the experimental vessel (symbols) and REFPROP 7.1 calculations (dashed lines). The 47.5% H_2 mixture (squares) was at ambient temperature. The 70% H_2 mixture (triangles) was immersed in ice-water (273 K).
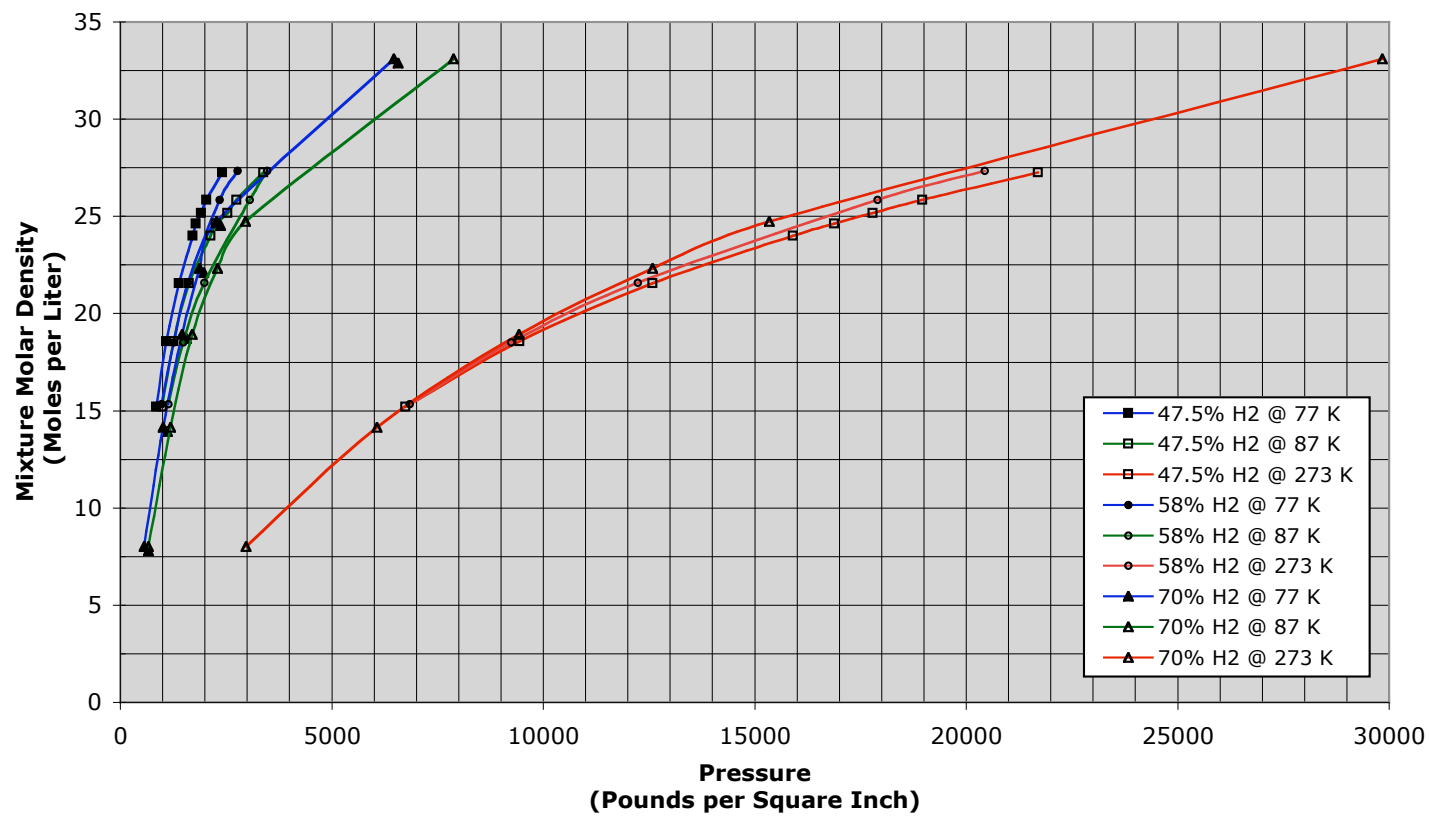
Figure 3. Molar density vs. pressure at 77 K (blue with solid symbols), 87 K (green), and 273 K (red) for hydrogen-nitrogen mixtures containing 47.5% H_2 (squares), 58% H_2 (circles), and 70% H_2 (triangles).
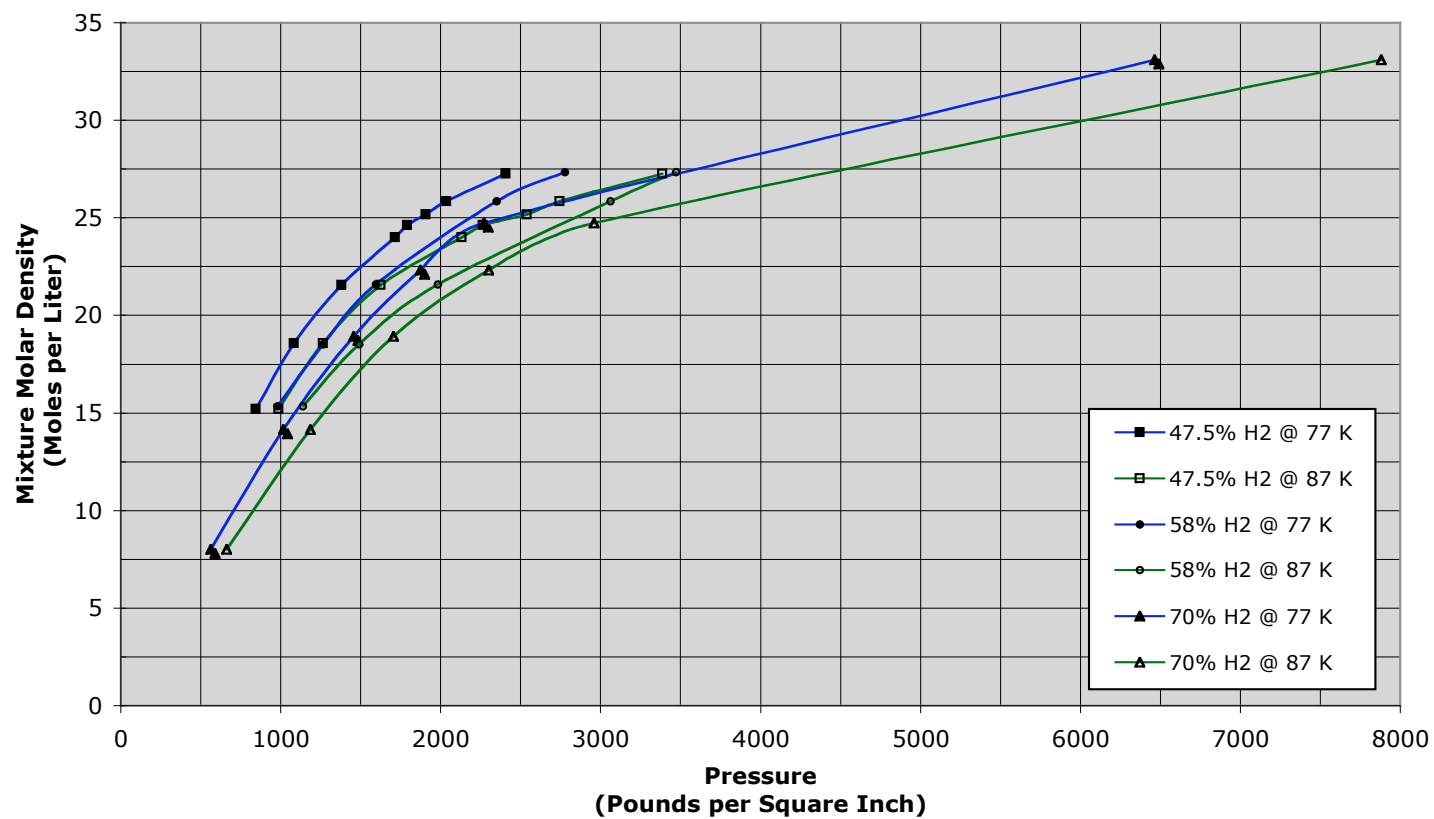
Figure 4. Molar density vs. pressure at 77 K (blue with solid symbols), and 87 K (green) for hydrogen-nitrogen mixtures containing 47.5% H_2 (squares), 58% H_2 (circles), and 70% H_2 (triangles).
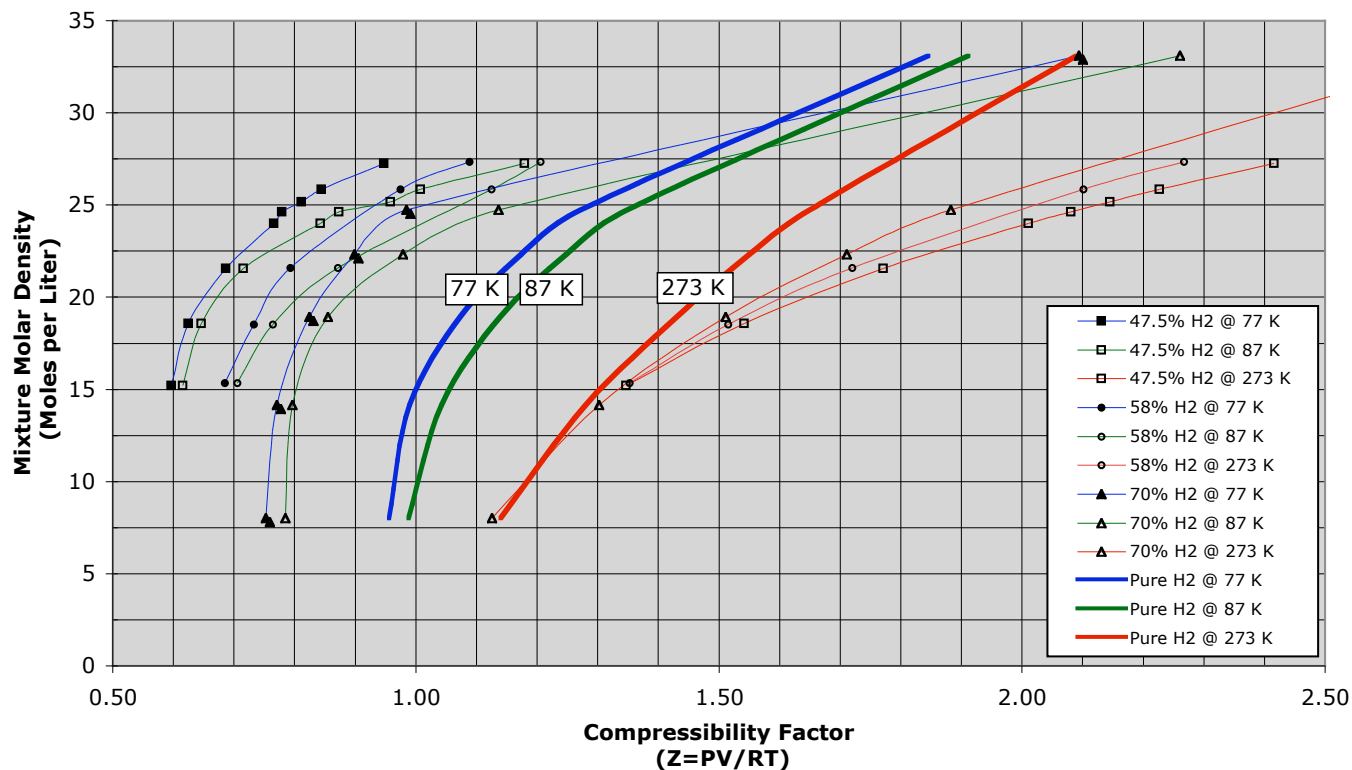
Figure 5. Molar density vs. compressibility factor (Z=PV/RT) at 77 K (blue with solid symbols), 87 K (green), and 273 K (red) for pure hydrogen gas (thick solid lines) and hydrogen-nitrogen mixtures containing 47.5% H_2 (squares), 58% H_2 (circles), and 70% H_2 (triangles). Mixtures are less compressible (higher Z) than pure H_2 at room temperature, but much more compressible (lower Z) than H_2 at cryogenic temperatures.
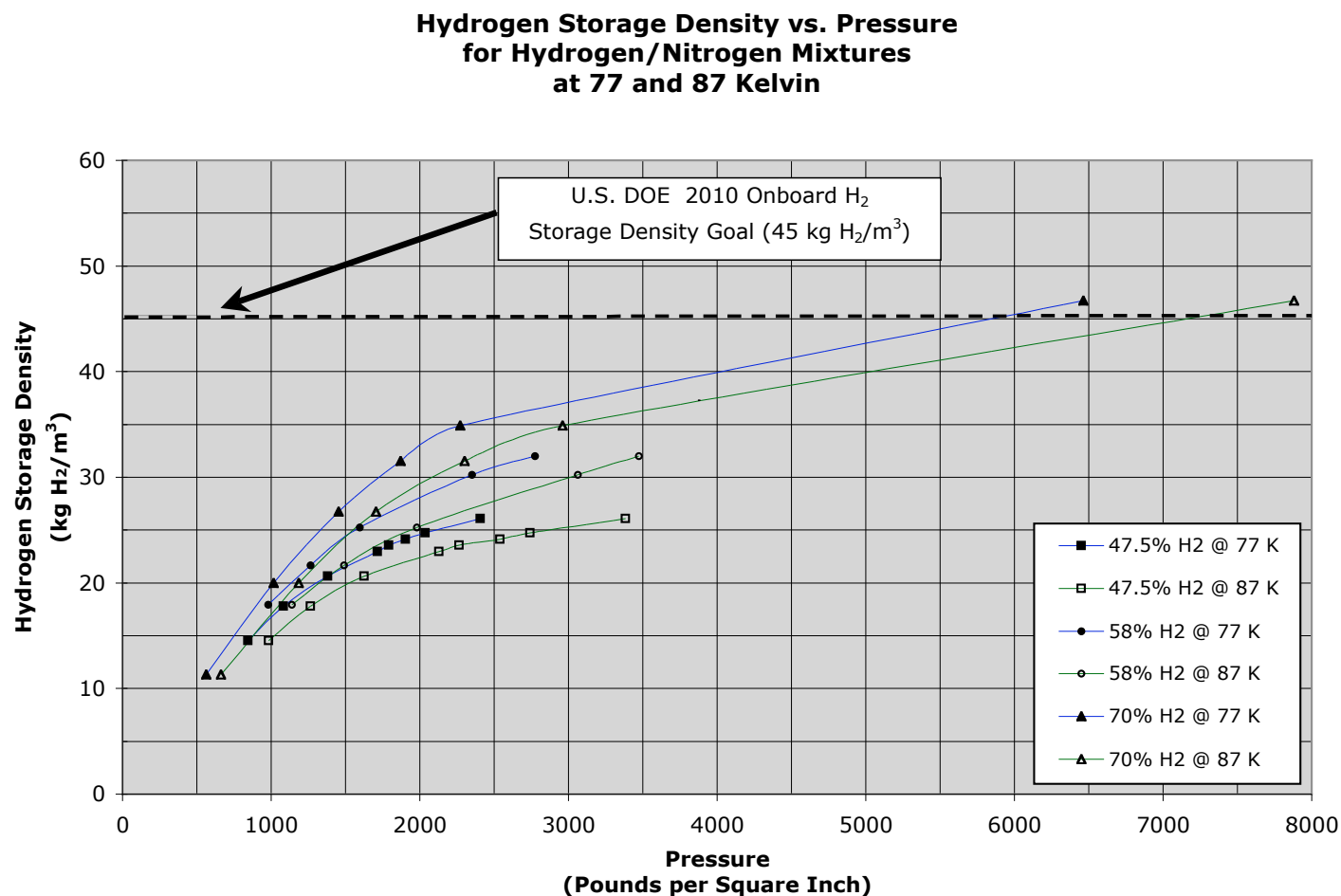
Figure 6. Hydrogen storage density vs. pressure at 77 K (blue with solid symbols), 87 K (green) for hydrogen-nitrogen mixtures containing 47.5% H_2 (squares), 58% H_2 (circles), and 70% H_2 (triangles). The 2010 U.S. Department of Energy onboard hydrogen storage goal was exceeded by a 70% H_2 mixture at pressures above 6000 psi and at 77 K.
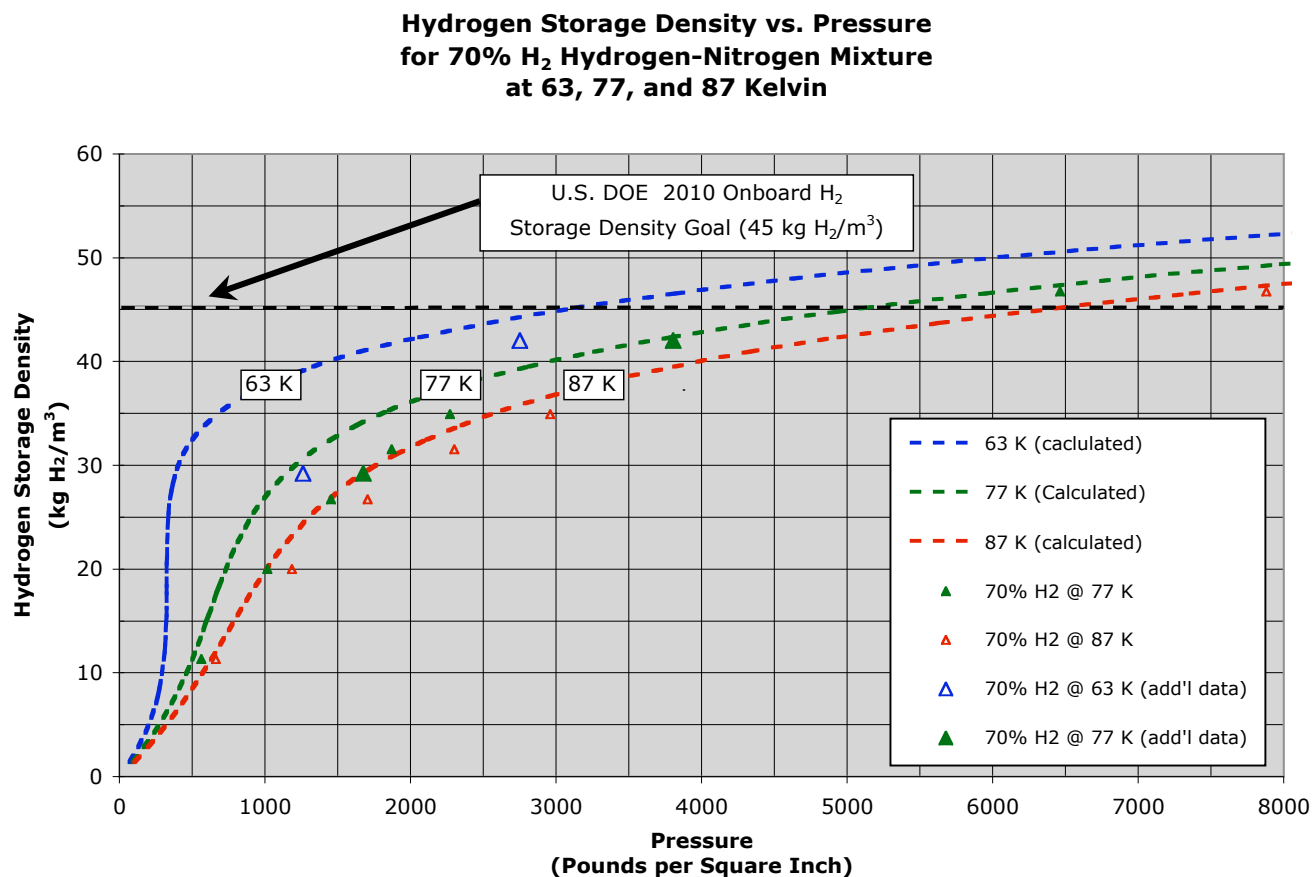
Figure 7. REFPROP calculations (dashed lines) of hydrogen storage density vs. pressure and experimental results (symbols) at 63 K (blue), 77 K (green with solid symbols), and 87 K (red) for a hydrogen-nitrogen mixture containing 70% H$_2$ (triangles). The larger solid triangles are experimental data in addition to those in Table 1. These data have somewhat greater experimental uncertainty, as they are were not conducted in tandem with ice-water and liquid argon measurements, and temperatures below 77 K were obtained by subcooling of LN$_2$ by vacuum pumping until solid N$_2$ formation was observed in the experimental vessel bath.

MATHESON TRI·GAS
Certified Mixture Grade

Matheson Tri-Gas Inc.
6775 Central Avenue
Newark, CA, 94560
Phone: 510-793-2559
Fax: 510-790-6241

TO: Univ of California
Lawrence Livermore Lab
7000 E. Ave
Livermore, CA 94550

PHONE:
FAX:

TO AVOID BACKFILL, CYLINDER PRESSURE MUST BE GREATER THAN PROCESS PRESSURE

SALES ORDER NUMBER: 293299
P.O. NUMBER: H366400 REL # KHQ0641
LOT NUMBER: 103-46-02543

PRODUCT: 30.0 pct Nitrogen UHP BAL Hydrogen UHP

CYLINDER NUMBER: RA-015542
SIZE: 3
CGA/DISS OUTLET: CGA 350
CONTENT: 35 cu. ft.
PRESSURE: 2000 psig

FILL DATE: May 13, 2004

COMPONENT	REQUESTED CONCENTRATION	BLEND TOLERANCE (+/-)	CERTIFIED CONCENTRATION	CERTIFICATION ACCURACY
Nitrogen	30.0 %	5 %	30.0 %	+/- 2%
Hydrogen	BAL		BAL	

TRACEABLE TO REFERENCE STANDARD SOURCE/NUMBER:
TRACEABLE TO NIST TRACEABLE WEIGHT CERTIFICATE: 822/260017-98

SPECIAL INFORMATION / ADDITIONAL COMMENTS

The product listed above and furnished under the referenced purchase order has been tested and found to contain the component concentration listed above. All values in mole/mole basis gas phase unless otherwise indicated. Matheson Tri-Gas Inc. warrants that the above product(s) conform at the time of shipment to the above description. Matheson Tri-Gas Inc. liability does not exceed the value of the product purchased.

Medi Senaki	Ex T8	May 19, 2004
ANALYST	SIGNATURE	DATE SIGNED

CALIBRATION DATA SHEET
PRESSURE TRANSDUCER

2900 MAIN ST ALAMEDA, CA 94501 PHONE (510) 522-9326 FAX (510) 522-3136

CUSTOMER	TSG JOB & ITEM #	CAL DATE	CAL DUE
LLNL HIGH PRESSURE LAB	2222	6/21/2004	6/21/2005

MFG.	MODEL	CAL BY	MFG S/N
TELEDYNE TABER	2403	R.K. STRAHL	915528

RANGE	UNITS	AMB. TEMP.	2ND S/N
30,000	PSIS	72.0	N/A

COND. AS RECIVED	COND. AS RETURNED	AMB PRESS.	EXITATION VOLTS dc
GOOD / IN TOL.	GOOD IN / TOL.	30.05	24.00

LINEARITY AND HYSTERESIS

SPAN	APPLIED PRESS.	S.L. SPAN VOLTS	OBS. VOLTS INC.	OBS. VOLTS DEC.	DEV. S.L. VOLTS INC.	DEV. S.L. VOLTS DEC.	VOLTS HYS.	% DEV. S.L. SPAN INC.	% DEV .S.L. SPAN DEC.	HYS.% SPAN
0%	0.00	0.008	0.008	0.007	N/A	N/A	-0.001	0.000	0.000	0.02
10%	3,000.00	0.503	0.508	0.503	0.005	0.000	-0.005	0.095	-0.004	0.10
20%	6,000.00	1.014	1.012	1.018	-0.002	0.004	0.006	-0.048	0.072	0.12
30%	9,000.00	1.518	1.522	1.520	0.004	0.002	-0.002	0.087	0.001	0.04
40%	12,000.00	2.021	2.012	2.014	-0.009	-0.007	0.002	-0.175	-0.135	0.04
50%	15,000.00	2.524	2.528	2.528	0.004	0.004	0	0.079	0.079	0.00
60%	18,000.00	3.027	3.020	3.029	-0.007	0.002	0.009	-0.143	0.079	0.18
70%	21,000.00	3.530	3.530	3.533	0.000	0.003	0.003	-0.008	0.052	0.06
80%	24,000.00	4.034	4.039	4.039	0.005	0.005	0	0.107	0.107	0.00
90%	27,000.00	4.537	4.533	4.534	-0.004	-0.003	0.001	-0.076	-0.056	0.02
100%	30,000.00	5.040	5.040	N/A	N/A	N/A	N/A			

REPEATABILITY / STANDARDS USED

% F.S. INC. PRESS.	RUN 1	RUN 2	RUN 3	MFG.	MODEL	S/N	RECAL	N.I.S.T #
0%	-0.003	-0.002	-0.002	AMETEK	DM-T-150	8681	1/8/2005	22036.001
50%	2.528	2.529	2.529	YOKOGAWA	3253-02	54KP0032	3/1/2006	YOKON/SN531
100%	5.040	5.042	5.043	FLUKE	8842A	4946290.000	10/9/2004	1-36671

SUMMARY

ZERO OFFSET (VOLTS)	0.008	END POINT LINEARITY %FSO (MFG SPEC <.25% FSO)	0.175
SPAN (VOLTS)	5.032	REPEATABILITY % FSO (MFG SPEC <.1% FSO)	0.060
SENSITIVTY (Mvolt/psi)	5.040	SHUNT CALIBRATION VALUE (VOLTS) @ AMB. PRESS	2.495
HYSTERESIS %FSO	0.009	SHUNT CALIBRATION VALUE (PSIS) @ AMB. PRESS	14,874.801

ERROR DISTRIBUTION

DEV. FROM ENDPOINT LINE (%FSO) vs PRESSURE (%FSO)

◆ % DEV. S.L. SPAN INC.   ■ % DEV .S.L. SPAN DEC.
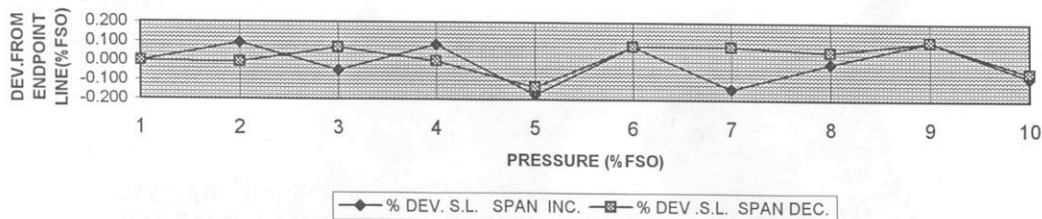
TECHNICAL SERVICES GROUP CERTIFIES THAT THIS INSTRUMENT HAS BEEN CALIBRATED TRACEABLE TO THE NATIONAL INSTITUTE OF STANDARDS & TECHNOLOGY AND CONFORMS TO ISO 10012 AND ANSI / NCSL Z-540. UNLESS OTHERWISE SPECIFIED MEASUREMENT UNCERTAINTIES ARE LESS THAN 1/4 OF TOLERANCE OR 1 MINOR DIVISION

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